OXIDATION OF ALLOYS FOR ADVANCED STEAM TURBINES

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Abstract

Ultra supercritical (USC) power plants offer the promise of higher efficiencies and lower emissions. Current goals of the U.S. Department of Energy's Advanced Power Systems Initiatives include coal generation at 60% efficiency, which would require steam temperatures of up to 760°C. This research examines the steamside oxidation of advanced alloys for use in USC systems, with emphasis placed on alloys for high- and intermediate-pressure turbine sections.

Introduction

For many years the temperatures and pressures of steam boilers and turbines were intentionally increased. These increases allowed for greater efficiencies in steam and power production, and were enabled by improvements in materials properties such as high temperature strength, creep resistance, and oxidation resistance. From 1910 to 1960, there was an average increase in steam temperature of 10°C per year, with a corresponding increase in plant thermal efficiency from less than 10% to 40%.[1] The first commercial boiler with a steam pressure above the critical value of 22.1 MPa (3208 psi) was the 125 MW Babcock & Wilcox (B&W) Universal Pressure steam generator in 1957—located at the Ohio Power Company's Philo 6 plant.[2] Since 1960 in the United States, the overall trend of increasing temperatures and pressures has stopped and stabilized at about 538°C and 24.1 MPa.[3] In Europe and Japan, where fuel costs are a higher fraction of the cost of electricity, temperatures and pressures continued to rise. An example of a state of the art power plant in Europe is the Westfalen (2004) plant, with steam conditions of 31.0 MPa/593°C/621°C.[4] It has a net plant efficiency of 43.5%, compared to 37% for a typical subcritical 16.5 MPa/538°C/ 538°C plant.[4] Today there is again interest in the United States for advanced supercritical power plants. Large increases in the cost of natural gas have led to the re-examination of coal power plants, and advanced supercritical plants offer advantages in lower fuel costs and lower emissions of SO_X, NO_X, and CO_2.[5] Table 1 shows four new advanced supercritical power stations at three sites that are either being proposed or constructed in the United States.[6] The steam conditions of the Council Bluffs plant is to be 25.4 MPa/566°C/593°C using a Babcock-Hitachi supercritical sliding pressure Benson boiler.[7]

Current U.S. Department of Energy research programs are aimed at 60% efficiency from coal generation, which would require increasing the operating conditions to as high as 760°C and 37.9 MPa. In general terms, plants operating above 24 MPa/593°C are regarded as ultra

supercritical (USC), those operating below 24 MPa as subcritical, and those at or above 24 MPa as supercritical (SC).[3]

Table 1 - New Advanced Supercritical Power Plant Starts in the United States.[6]

Location	Size	Status	In Service	Cost	Fuel
Council Bluffs Iowa	790 MW	Construction started 8/2004	2007	1.2 B$	Coal
Trimble County Kentucky	750 MW	Proposed 11/2004	2010	1.2 B$	Illinois Basin Coal
Oak Creek Wisconsin	600 MW (2)	In Development 12/2004	2009-2010	2.5 B$	Powder River Basin Sub-Bituminous

In the past thirty years, advances in the high temperature strength of ferritic steels have allowed for the increase of operating temperatures and pressures, but without the thermal fatigue issues of the austenitic steels that had to be used to obtain the required high temperature strengths in the early 1960s. Ferritic steels, as used here, refers to the equilibrium structure. In practice, a martensitic or partially martensitic structure is obtained from heat-treating. The upper temperature for use of ferritic steels appears to be limited to about 620 to 630°C. For temperatures above 630°C, the most promising candidate alloys are nickel-base superalloys.

The purpose of this paper is to report on research that examines the steamside oxidation of advanced alloys for use in supercritical turbine systems. Low-pressure turbine sections of USC systems would not be expected to differ from current designs, so emphasis is placed on alloys for high- and intermediate-pressure turbine sections. Initial results are presented along with a review of potential materials for supercritical turbines.

Benefits

The driving force for increased operating temperatures and pressures has been increased efficiency in power generation. Recently, an additional recognized benefit has been decreased CO_2 emissions. Estimates of the cost effectiveness of various ways to improve the efficiency of power plants are shown in Table 2. Table 2 shows that increasing the steam temperature is one of the more cost effective ways of increasing efficiency, while increasing the steam pressure is less effective.

For reduced CO_2 emissions, calculations by Booras *et al.*[8] indicate that a subcritical 37% efficient plant 500 MW plant burning Pittsburgh #8 coal would produce about 850 tons of CO_2 per kWh. Ultra supercritical plants at 43% and 48% efficiency would respectively produce about 750 and 650 tons of CO_2 per kWh.

OBJECTIVES

The objectives of this project are to:

- Assist in the development of high strength austenitic or nickel-base superalloys for use in USC steam turbines by conducting steam-side corrosion tests on candidate materials.

Table 2 - Cost effectiveness of methods to improve fossil fuel power plant efficiency.[3] Cost is in terms of millions of U.S. Dollars per net percent increase in lower heating value (LHV) efficiency.

Rank	Method	Cost
1	Reducing condenser back pressure	3.1
2	Increase to 8th extraction point feed water heater, raising feed water temperature	3.8
3	Raising live steam and reheat temperatures	8.3
4	Raising live steam temperature	8.6
5	Using separate boiler feed pump turbine (BFPT) instead of main turbine driven pump	9.6
6	Raising live steam pressure	25.1
7	Change from single to double reheat	38.2
8	Using separate BFPT condenser	41

- Determine the effect of pressure on the corrosion process.
- Examine curvature effects on spallation.

The research aims to bridge the gap in information between the various steam conditions to study the resistance of target alloys and the role of pressure in the corrosion mechanisms. The alloys to be examined include the ferritic alloy SAVE12, the austenitic alloy SUPER304H, the high Cr and high Ni alloy HR6W, and the nickel-base superalloys Inconel 617, Inconel 718, Inconel 740, Haynes 230, M-252, Nimonic 90, and Refractory 26.

The experimental work was to consist of three types of tests:

Supercritical Steam: Long-term tests at the supercritical steam temperatures and pressures. Test durations of 3000 hours with three 1000 hr segments. Conditions as high as 760°C (1400°F) and 37.9 MPa (5500 psi).

TGA in Steam: Experiments using thermal gravimetric analysis (TGA) with steam and argon at atmospheric pressure. This will test alloys for susceptibility to steam oxidation using relatively short test durations (~300 hr) and obtain kinetics parameters.

Cyclic Oxidation: Experiments using cyclic oxidation tests in air in the presence of water vapor. This will test the adhesion and spallation behavior of the protective oxides that form on the test alloys. The effect of sample curvature will be examined in selected systems.

Furnace Exposure: Experiments that expose alloys in moist air (3% H2O) for long exposure times at temperatures from 650 to 800 °C. These are economical tests that bridge the gap between exposure in air and exposure in steam.

TECHNICAL PROGRESS

Alloys

The nominal compositions of alloys selected for testing are given in Table 3. These include a subset of the alloys examined by the Advanced Power System Initiative on USC boilers,[9] which

are the ferritic alloy SAVE12, the austenitic alloy SUPER304H, the high Cr and high Ni alloy HR6W, and three nickel-base superalloys Alloy 617, Alloy 230, and Alloy 740. All represent the highest high-temperature strength alloys in their respective alloy classes. Also included are four superalloys identified[10-11] as candidates for blade materials for USC conditions: Alloy M-252, Refractory 26, Nimonic 90, and Alloy 718. The last two alloys listed in Table 3, Alloys J1 and J5, were produced for alloy development into solid oxide fuel cells.[12] However, Alloy J1 is an equivalent composition to Mitsubishi alloy LTES700, a low coefficient of thermal expansion nickel-base alloy developed for use as fasteners and blades in both current and USC steam turbines.[13] Alloy J5 is a modified version of J1.

Table 3 - Nominal compositions of alloys of interest for USC turbine applications. Reference numbers follow the alloy name.

Alloy	Fe	Cr	Ni	Co	Mo	Nb	C	Si	Mn	Ti	Al	Other
SAVE12[14]	Bal	11		3		0.07	0.1	0.3	0.2			3 W 0.2 V 0.04 Nd 0.04 N
SUPER304H[14] SUS304J1HTB	Bal	18	9			0.4	0.1	0.2	0.8			3 Cu 0.1 N
HR6W[14]		23	43			0.18	0.08	0.4	1.2	0.08		6 W 0.003 B
Alloy 617[15] ASME Code 1956		22	55	12.5	9		0.07				1	
Alloy 230[15] ASME Code 2063 UNS N06230	<3	22	55	<5	2		0.1				0.35	14 W <0.015B 0.02 La
Alloy 740[9]	1	24	49	20	0.5	1.8		0.5		1.6	0.75	
M-252[15]	0.75 max	19	56.5	10	10		0.15			2.6	1	0.005 B
Refractory 26[15]	16	18	38	20	3.2		0.03			2.6	0.2	0.015 B
Nimonic 90[15] UNS N07090	1.5	19.5	55.5	18			0.06			2.4	1.4	
Alloy 718[15] UNS N07718	18.5	19.0	52.5		3	5.1	0.08 max			0.9	0.5	0.15 Cu max
J1[12]		12.1	Bal		18					1	0.8	
J5[13]		12.5	Bal		22				0.5	1		0.04 Y

The alloys used in initial tests included SAVE12, HR6W, Alloy 617, Alloy 230, Alloy 740, Alloy 718 and Nimonic 90. There are two versions of the SAVE12 alloy (nominally 11Cr) that were examined: a version that is corrosion resistant to higher temperatures (10.5Cr), and a higher strength version (9.5Cr). The surface on all samples (except where noted) was a polish to 600 grit. Curvature effects were examined on SAVE12 (10.5Cr) and HR6W by machining samples from thick walled pipe. Each of the curvature samples have one curved surface, representing either the inside (concave) or outside (convex) surface of the pipe, Fig. 1. The curved surfaces were machined from as-received pipe (to remove mill-scale) and not subsequently polished to 600 grit.

The alloys in Table 3 not used in initial tests (SUPER304H, M-252, and Refractory 26) have not been examined due to a lack of alloy material from which to make samples.

Fig 1 – Section of SAVE12 10.5Cr pipe (2-in O.D.) and curvature samples cut from pipe. All but one side of the curvature samples are flat. Samples: concave (left) and convex (right).

Supercritical Steam

A test loop in supercritical steam with temperatures and pressures up to 760°C (1400°F) and 37.9 MPa (5500 psi) was planned as shown in Fig. 2. The feed water system, shown in Fig. 3, allows for measurement and some control of water chemistry (pH, dissolved oxygen (DO), and conductivity). Tests would be done to represent an oxygenated system with 150-200 ppb DO and a pH of 9.2 to 9.6.

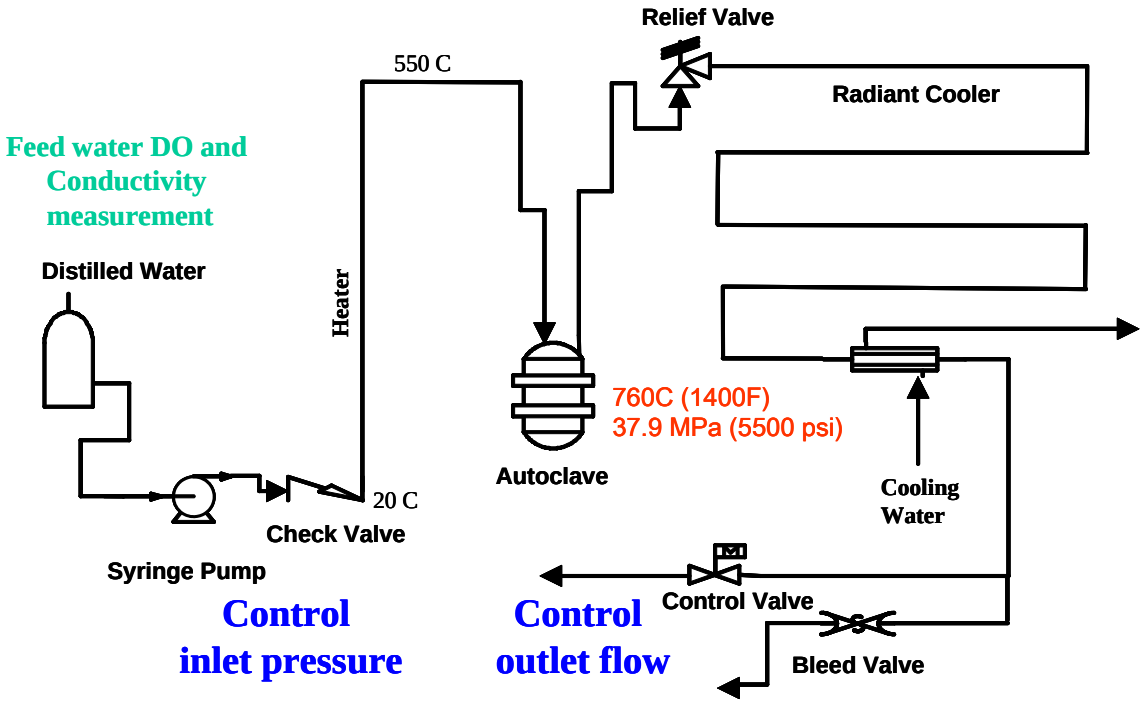


Fig 2 – Supercritical test loop for exposures in supercritical steam with temperatures and pressures up to 760°C (1400°F) and 37.9 MPa (5500 psi). Autoclave size of 1 liter.

The autoclave was due for delivery on 12/31/2004. It has not arrived as of the date of this report in May 2005. Negotiations are underway to obtain the autoclave. The feed water system and most of the other components have been obtained.

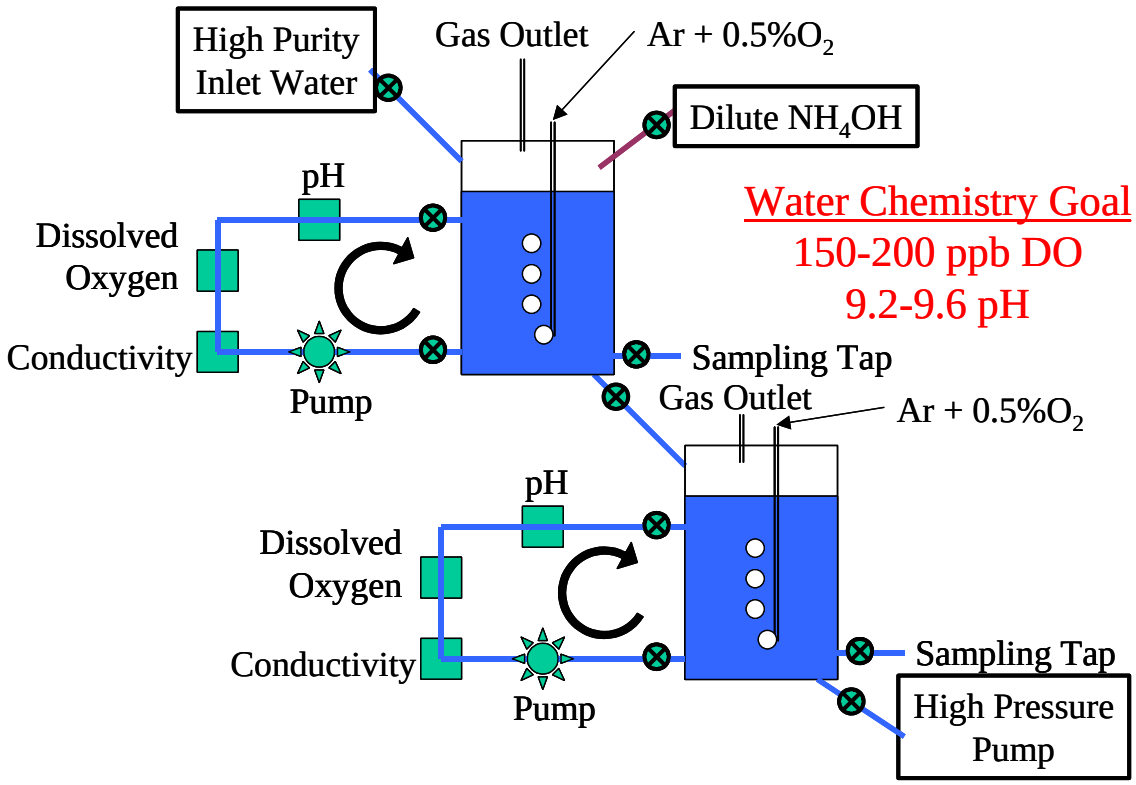


Fig 3 – Feed water system for supercritical steam exposures.

Cyclic Oxidation

Cyclic oxidation experiments were conducted in air in the presence of steam at atmospheric pressure. This was designed to examine the adhesion and spallation behavior of the protective oxides that form. The tests consisted of 1-hour cycles of heating and cooling (55 minutes in the furnace and 5 minutes out of the furnace) in a tube furnace equipped with a programmable slide to raise and lower the samples, Fig. 4. Water was metered into the bottom of the furnace along with compressed air (50% water vapor-50% air, by volume). The exposure temperature for these initial tests was 760°C. Both flat and curvature samples were examined.

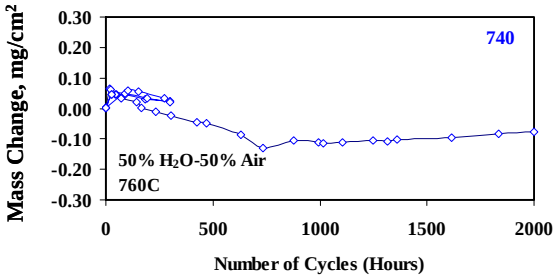
Fig 4 – Cyclic oxidation apparatus for testing in atmospheric pressure steam/air mixtures at up to 800°C.

Figures 5-6 show results for Alloys 740 and 230, respectively. Over 2000 hours, both alloys had a net mass loss and very thin oxide scales.

Figures 7-8 show results for Alloys 617 and 718, respectively. Over 2000 hours, Alloy 617 had virtually no net mass loss and a very thin oxide scale. Alloy 718 has only been tested for 300 hrs

and shows much the same results as Alloys 740, 230, and 617 (Figs. 5-7) for this time period—a small increase in mass.

A



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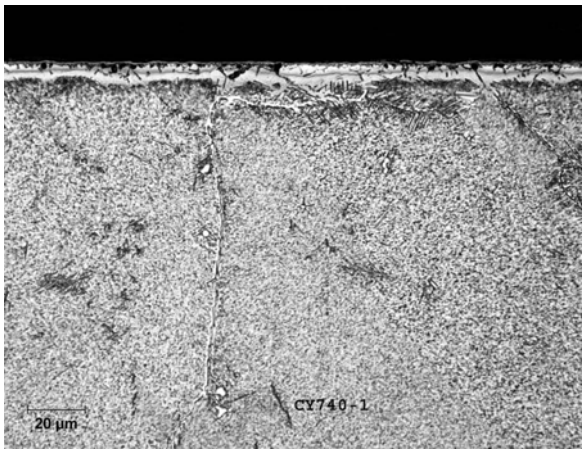
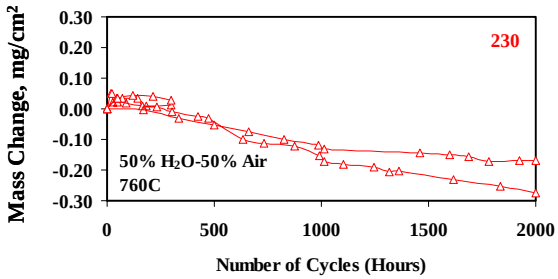


Fig 5 – A) Cyclic oxidation results for Alloy 740 at 760°C in 50% H_2O-50% air with hourly cycles. B) Light microscopy after 2000 hr. C) Light microscopy cross-section after 2000 hr (etched).

A



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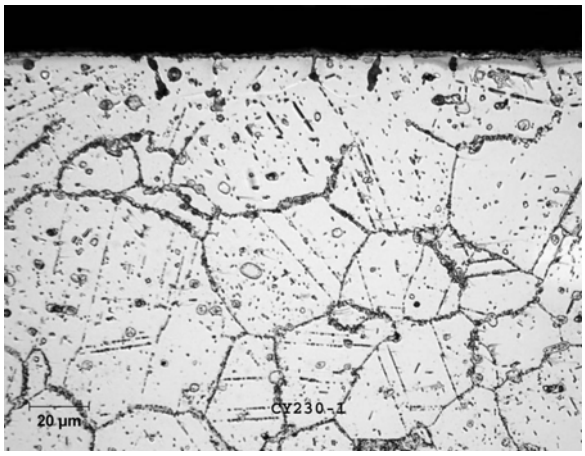
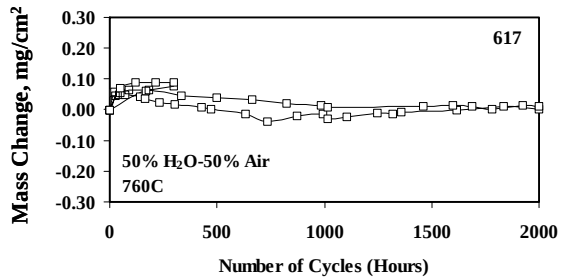


Fig 6 – A) Cyclic oxidation results for Alloy 230 at 760°C in 50% H_2O-50% air with hourly cycles. B) Light microscopy after 2000 hr. C) Light microscopy cross-section after 2000 hr (etched).

A



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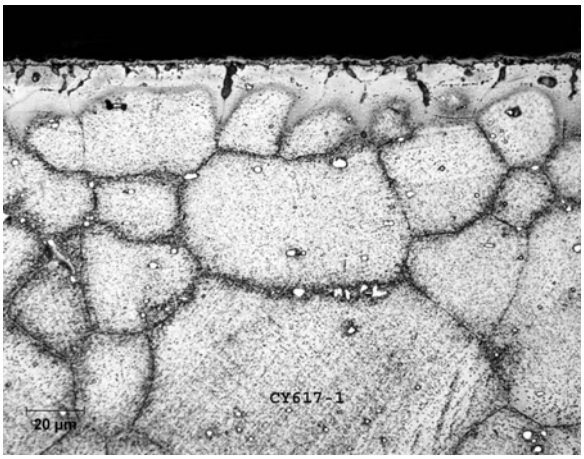
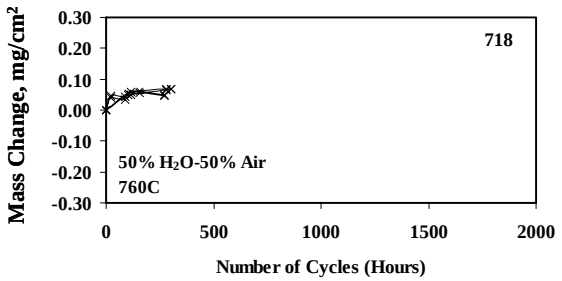


Fig 7 – A) Cyclic oxidation results for Alloy 617 at 760°C in 50% H_2O-50% air with hourly cycles. B) Light microscopy after 2000 hr. C) Light microscopy cross-section after 2000 hr (etched).

A



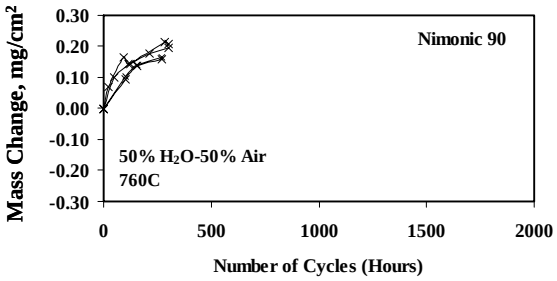
B

Fig 8 – A) Cyclic oxidation results for Alloy 718 at 760°C in 50% H_2O-50% air with hourly cycles. B) Light microscopy after 2000 hr.

Figures 9-10 show results for Nimonic 90 and HR6W, respectively. Over 300 hours, Nimonic 90 has shown more mass gain than other materials (Figs. 5-8). Over 2000 hours, HR6W has

shown a net mass increase. The curvature sample curves in Fig. 10 are closely following the flat sample (for the first 300 hours).

A



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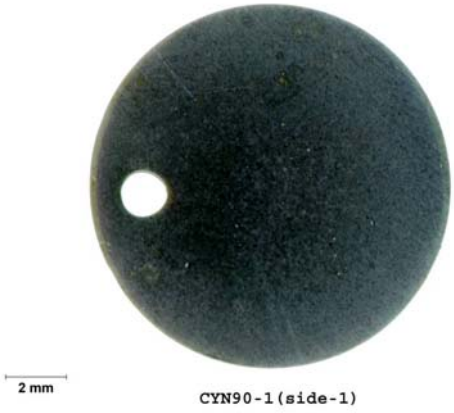
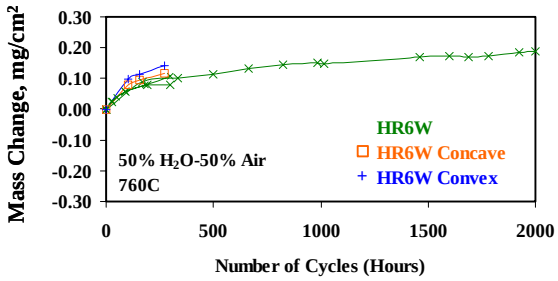


Fig 9 – A) Cyclic oxidation results for Nimonic 90 at 760°C in 50% H_2O-50% air with hourly cycles. B) Light microscopy after 2000 hr.

A



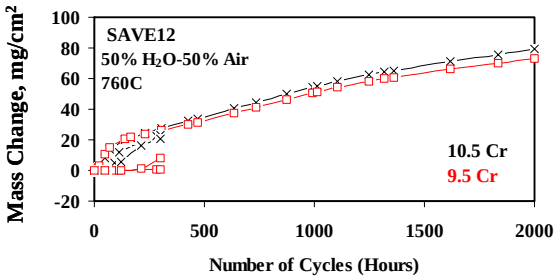
B

Fig 10 – A) Cyclic oxidation results for HR6W at 760°C in 50% H_2O-50% air with hourly cycles. B) Light microscopy after 2000 hr.

Figures 11-12 are for SAVE12. Figure 11A compares the 9.5 Cr and 10.5 Cr versions of SAVE12. The test temperature of 760°C is far above the use temperature of SAVE12, as indicated by the large oxidation rates (as well as being at a temperature where SAVE12 lacks sufficient creep strength). Under these unprotective conditions, the 9.5 Cr version shows slightly less mass gain than the 10.5 Cr version. The lack of reproducibility in the initial oxidation behavior is also shown in Fig. 11. Figure 11C shows that the scale is much thicker than in the nickel base alloys (that also have higher Cr levels).

Figure 12 shows the results for curvature samples. The higher mass gain of the convex sample is probably due to the lack of reproducibility in initial oxidation. The slopes are similar after 600 hours. Figures 12B and 12C show spallation of the curvature samples in a greater degree than for the flat sample in Fig. 11B.

A



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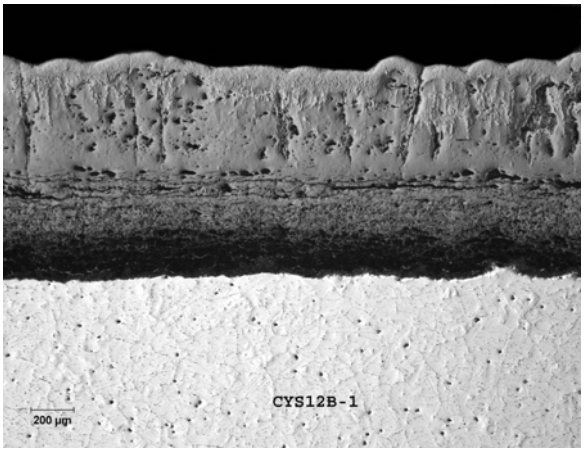

Fig 11 – A) Cyclic oxidation results for SAVE12 at 760°C in 50% H_2O-50% air with hourly cycles. B) Light microscopy for 10.5 Cr after 2000 hr. C) Light microscopy cross-section for 10.5 Cr after 2000 hr (etched).

A

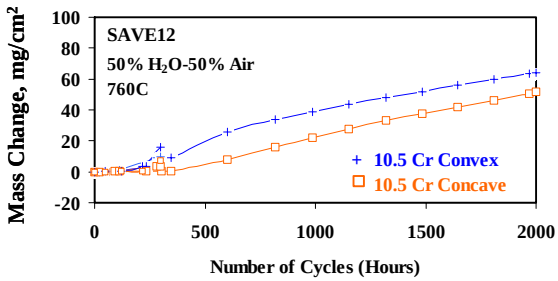

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Fig 12 – A) Cyclic oxidation results for concave and convex SAVE12 (10.5 Cr) at 760°C in 50% H_2O-50% air with hourly cycles. B) Light microscopy for concave 10.5 Cr after 2000 hr. B) Light microscopy for convex 10.5 Cr after 2000 hr.

Thermogravimetric Analysis (TGA)

Experiments were conducted using thermogravimetric analysis (TGA) with steam at atmospheric pressure. This was designed to obtain information on oxidation kinetics using relatively short (300 hr) test durations. The TGA tests consist of suspending a sample from a Cahn D-101 microbalance in flowing steam for 300 hours at a constant elevated temperature (650-800°C), Fig. 13. Steam is generated by injecting a metered amount of O_2-saturated water into heated tubing to supply a minimum flow rate of 2 mm/s of steam in the reaction chamber. Initial experiments used pure steam. More recent tests used a carrier gas of 60% Ar along with the steam.

Fig 13 – TGA apparatus for testing in atmospheric pressure steam at up to 800°C.

Table 4 summarizes the results of the 300 hr TGA tests in O_2-saturated steam plus 60%Ar at 800°C. The reaction order and parabolic R^2 are measures of how well the data fit parabolic kinetics of

$$\text{Mass change} = k_p t^{1/n}$$

where k_p is the parabolic rate constant, t is time, and n is the reaction order (n = 1 for linear kinetics and n = 2 for parabolic kinetics). The parabolic R^2 measures how well the data correlate with parabolic behavior using the calculated k_p (with 1 being exact correlation and 0 being no correlation). The parabolic R^2 values for SAVE12 and one of the Alloy 617 tests were quite close to 1, showing excellent correlation with parabolic kinetics and with relatively little noise in the data. The oxidation rates of Alloy 230, HR6W, Alloy 740, and one of the Alloy 617 tests were lower, and with more noise in the mass change data, which resulted in lower parabolic R^2 values.

Table 4 – Thermogravimetric analysis (TGA) for 300 hr tests in O_2-saturated steam plus 60%Ar at 800°C

Alloy	%Cr	Reaction Order, n	Parabolic R^2	Parabolic Rate Constant, k_p $mg^2cm^{-4}s^{-1}$
SAVE12 9.5Cr	9.5	1.78	1.000	1.4×10^{-3}
SAVE12 10.5Cr	10.5	1.70	1.000	1.9×10^{-3}
SAVE12 10.5Cr	10.5	1.76	0.995	2.0×10^{-3}
J1	12.1	1.73	0.990	3.8×10^{-7}
J5	12.5	1.91	0.990	1.7×10^{-7}
Alloy 617	22	1.62	0.960	1.4×10^{-7}
Alloy 617	22	2.63	0.585	3.9×10^{-8}
Alloy 230	22	1.78	0.878	6.9×10^{-8}
Alloy 230	22	1.79	0.645	3.7×10^{-8}
HR6W	23	1.87	0.524	3.8×10^{-8}
Alloy 740	24	2.20	0.527	2.0×10^{-7}

The increase in noise is due to limitations in the TGA system. Figures 14-15 show these differences. In Fig. 14, the curve for SAVE12 (10.5 Cr) is quite smooth. In Fig. 15, the curve for HR6W is quite noisy—enough to make identification of spalls unclear. Future tests at lower temperatures will probably be restricted to alloys that oxidize faster, such as the SAVE12 alloys and possibly SUPER304H.

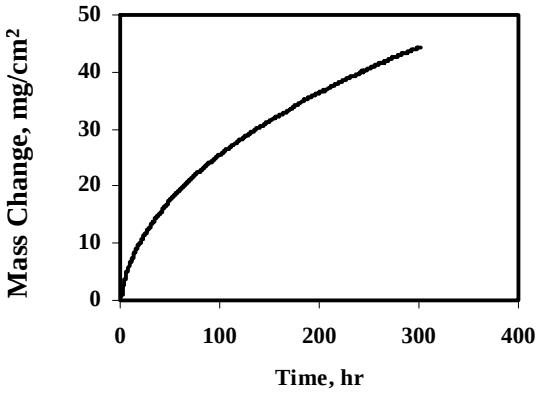

Fig 14 – TGA results for SAVE12 10.5Cr in O_2-Ssaturated steam plus 60%Ar at 800°C.

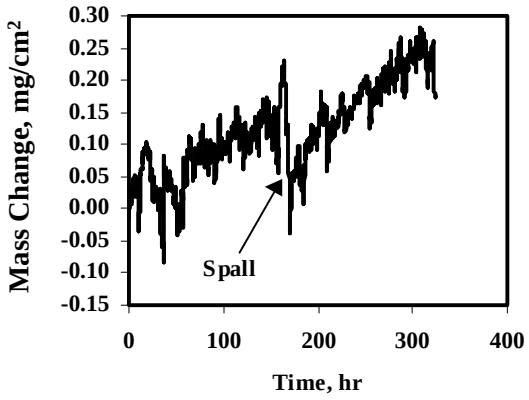

Fig 15 – TGA results for HR6W in O_2-Ssaturated steam plus 60%Ar at 800°C.

Furnace Exposures

Experiments were added that exposed samples to moist air at atmospheric pressure. These tests consisted of exposing the samples to air that was bubbled through water, resulting in up to 3% water vapor in the atmosphere. Samples were periodically removed from the furnace for mass measurements, and then replaced in the furnace for further exposure. More recent tests have attempted improve the procedures by ensuring that the input air is water saturated (by bubbling though two long columns of water) and that the temperature cycles are more tightly controlled (100 hr cycles with 200 °C/hr ramp rates).

The results for furnace exposures in moist (3% H_2O) air are shown in Fig. 16 at 700°C and Figs. 17-18 at 800°C. The oxidation rates for the ferritic steel SAVE12 at 800°C (Fig. 17) were quite high, with 10.5Cr having slightly higher rates than 9.5Cr. At 700°C (Fig. 16), the oxidation rates of SAVE12 were much lower; with 10.5Cr showing much less mass increase than 9.5Cr.

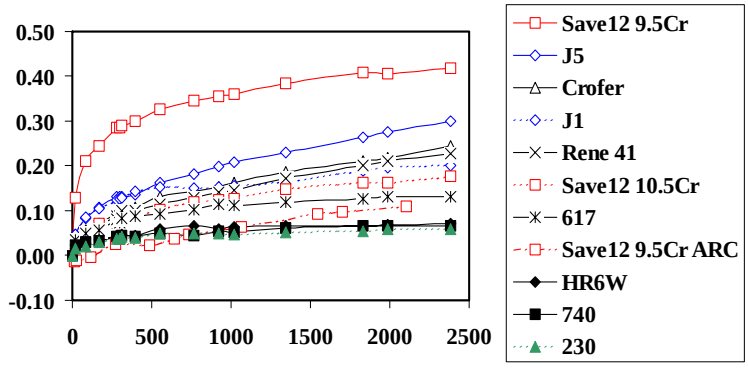

Fig 16 – Furnace exposures at 700°C in moist (3% H_2O) air.

This is similar to the reported difference between 9% Cr and 12% Cr steels becoming significant at 650°C.[16-17] All of the nickel base alloys had modest mass gains at 700 °C (Fig. 16) and 800°C (Fig. 18).

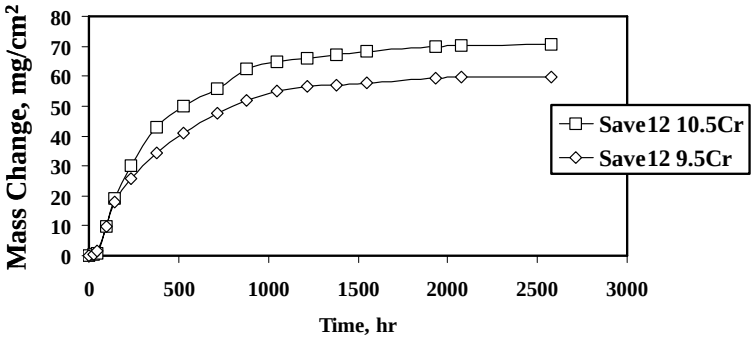

Fig 17 – Furnace exposures of SAVE12 at 800°C in moist (3% H_2O) air.

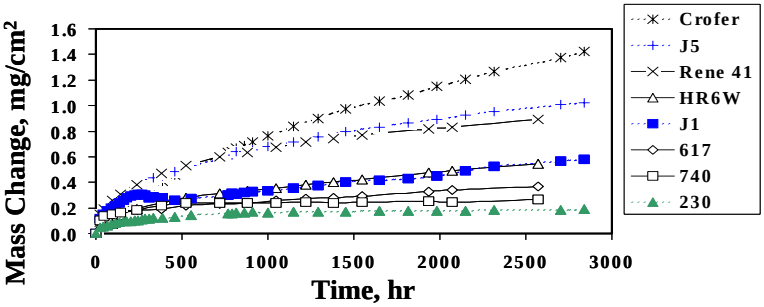

Fig 18 – Furnace exposures of nickel-base alloys at 800°C in moist (3% H_2O) air.

SUMMARY

The status of research to examine the steamside oxidation of advanced alloys for use in supercritical systems was presented. The alloys of interest were mainly nickel-base superalloys, the exceptions being selected high-strength ferritic (SAVE12) and austenitic (SUPER304H) stainless steels. The initial results from cyclic oxidation in moist air at 760°C, TGA in steam plus 60% Ar at 800°C, and furnace exposures in moist air at 700°C and 800°C were described:

- Cyclic oxidation of nickel-base superalloys indicated a relatively steady rate of oxide scale loss after an initial mass gain.
- Cyclic oxidation of the ferritic SAVE12 alloys resulted in high oxidation rates with linear kinetics after an initially higher oxidation rate.
- Cyclical oxidation of convex and concave SAVE12 with 10.5Cr and HR6W need further examination.
- TGA tests resulted in measured parabolic rate constants on the order of 10^{-3} $mg^2cm^{-4}s^{-1}$ for the SAVE12 alloys, and 10^{-7} to 10^{-8} $mg^2cm^{-4}s^{-1}$ for Alloy 617, Alloy 230, Alloy 740 and HR6W.

- Furnace exposures gave quite high oxidation rates for the ferritic steel SAVE12 at 800°C, with 10.5Cr having slightly higher rates than 9.5Cr. At 700°C, the oxidation rates of SAVE12 are much lower; with 10.5Cr showing much less mass increase than 9.5Cr.

The major problem has been the failure of the autoclave to arrive on its delivery date of 12/31/2004. It has not arrived as of the date of this report in May 2005. Negotiations are underway to obtain the autoclave. This has severely limited progress on determining the effects of pressure on oxidation.

A minor problem has been sensitively limitations of the TGA apparatus. This limitation is apparent in tests with very low oxidation rates. Therefore, future TGA tests will be limited for nickel-base superalloys (that have very low oxidation rates).

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